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Jet fuel deposits, some of them supplied on their original support by Dr. Hazlett of the Naval Research Laboratory were examined by attenuated total reflection spectroscopy and by infrared Fourier transform emission spectroscopy for the purpose of finding a procedure suitable for in situ analysis. The infrared radiation emitted by samples at 50-65°C was adequate for qualitative analysis, which can readily be made quantitative by further work. Definite sharp peaks corresponsing to aromatic materials were found.

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DETERMINATION AND ANALYSIS
OF JET AND MISSILE FUEL DEPOSITS

by

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I. SUMMARY

Jet fuel deposits, some of them supplied on their original support by Dr. Hazlett of the Naval Research Laboratory were examined by attenuated total reflection spectroscopy and by infrared Fourier transform emission spectroscopy for the purpose of finding a procedure suitable for in situ analysis. The infrared radiation emitted by samples at 50 - 65°C was adequate for qualitative analysis, which can readily be made quantitative by further work. Definite sharp peaks corresponding to aromatic materials were found.

II. INTRODUCTION

Jet fuel will form deposits in fuel lines and manifolds subject to relatively high temperatures. These deposits reduce the efficiency of heat transfer, narrow orifices and otherwise interfere with Naval Aircraft operation. The quantities of deposits formed depend on fuel type and operating conditions, but the nature of the deposits has usually been described as "coke". Coke or "carbon" has hardly been chemically identified although the literature is replete with mechanisms of its formation. The "coke" is usually solidly bound to the surface on which it is formed: its removal by scraping and subsequent solvent extraction may not result in proper molecular identifications especially since the coke is unlikely to be of homogeneous composition, (although elemental analysis is, of course, possible and is usually performed).

Our procedures were designed to analyze the molecular composition of the coke in a non-destructive way, i.e. without removal of the deposit from the support. We applied infrared emission and reflection spectroscopy, of necessity to the outermost deposit layer. Unless this layer is very thin--which it was not for the samples studied--it represents the latest material laid down and is uninformative on the surface - to - carbon bonding. On the other hand, it should be the deposit layer most closely associated with the composition of the fuel.

Some sharp emission peaks corresponding to a large aromatic content were found. Since we expected, naively perhaps, only blackbody emission from black, irregular deposits, we were surprised. The results are therefore very interesting and deserve further attention.

III APPARATUS AND PROCEDURE

3.1 Reflectance Techniques

Since our infrared Fourier interferometer was only intermittently available, we used our Perk ..-Elmer Model 221 Grating Spectrophotometer for this feasibility study. Our Harrick VRA attachments for either attenuated total reflection (ATR) or specular reflection studies (the specular reflection attachment is an attachment to the VRA) were designed for the Perkin-Elmer spectrophotometer and could therefore be readily put into service. Figure 1 shows the arrangement for ATR spectroscopy and Figure 2 for specular and diffuse reflectance. Both internal and external reflectance can be used dependent on whether a germanium or KRS-5 hemicylinder is put in contact with the sample surface or not. In the former instance spectra of better quality are possible when the refractive indices of sample and hemicylinder are appropriately matched. The theories of these procedures are well described in Harrick's book and need not be explained here.

None of the internal reflectance procedures, i.e. germanium or KRS-5 plates or hemicylinders were successful with any of the deposits, not even the smooth-appearing carbon on the inside of a fuel-line tube (which was flattened for this purpose). The "carbon" surfaces were too rough for proper contact with the crystal surface. On the other hand, the fuel-line deposit mentioned was smooth enough to give a good external reflectance spectrum.

The well-known mull (suspension in Nujol) and potassium bromide suspension techniques were also used; the results were poor because of excessive scattering. Some scraped deposits were very finely ground in a mortar prior to their suspension, recognizing that the samples could thereby be seriously altered. However, even these attempts failed.

With more time and effort a measure of success could probably have been achieved in the form of identifiable spectra. However, since our aim was directed primarily toward procedures for in place materials, such further work could not be justified.

3.2 Infrared Emission

Our infrared emission Fourier microspectrophotometer turned out to be the best approach to the problem. Since it was described in detail in a recent publication, which is appended to this report, it need not be discussed much further here. The analysis of the deposits proved to be much easier than the analysis of the lubricating oils in operating bearings, for which our spectrometer was designed; no moving parts were involved and larger sample surfaces were available. It was necessary to heat the samples on their support to 50° to 65°C by a cartridge heater to provide an adequate temperature differential between source and detector. This temperature should not be high enough to cause chemical change; however,

future work along these lines should be carried out in a nitrogen atmosphere, a perfectly feasible approach although it should be mentioned that the nitrogen should be preheated to the sample temperature to avoid a temperature gradient through the sample.

The sensitivity of our Fourier instrument for emission exceeds that of commercial Fourier spectrophotometers by at least an order of magnitude for these reasons: (i) our instrument has four-inch diameter optics throughout, whereas one-or two-inch optics are the best commercial practice, (ii) our special inlet optics, containing a high-aperture Cassegrainian objective, collects the radiation over a wide solid angle, (iii) a blackbody reference source with phase-sensitive detection by a Golay detector provides for a high signal/noise ratio with weak sources, and (iv) the software we built into our dedicated minicomputer contains batteries of numerical filters and other sophisticated mathematics to reduce random noise to a minimum. Because of these unique features, scheduling has become a problem. Another instrument might be built to reduce the work load and to provide a backup system in case of failure.

IV RESULTS

4.1 Reflection Spectra

Jet fuel deposit #AFFB-12 (specimen D, see Table I for a summary of deposit samples) was run by specular reflection as described in the previous section. For this purpose the tube on which the deposit had accumulated was slit open, flattened, and a 0.5 x 0.5 inch square section was cut and inserted into the holder of the Harrick attachment. The attachment was placed into the sample beam, while an attenuator was put into the reference beam of the Perkin-Elmer grating spectrophotometer to nearly balance the energy throughput from the common source.

Figure 3a shows a broad maximum at about 3480 cm^{-1}, which corresponds to bonded OH··O, either alcoholic or phenolic. Since no other features are evident in this spectral region, the phenolic assignment is more likely. Figure 3b represents the low frequency range of the same spectrum. The 1400-1800 cm^{-1} region is unfortunately obscured by atmospheric water vapor and carbon dioxide absorption (the instrument can be surrounded by dried and carbon dioxide-free air, but this step was not undertaken for this preliminary work). However, any significant carbonyl peak (near 1750 cm^{-1} is clearly absent. No features were resolved in the 700-900 cm^{-1} hump.

Other samples were tried by reflection, but with no reportable results. The energy of the reflected and scattered radiation was very weak, so that the instrument had to be pushed to its limit. The Fourier spectrometer would have been more sensitive for reflection, but was not used (i) for lack of time and (ii) because the emission spectra turned out quite well.

4.2 Emission Spectra

The same sample (Jet fuel deposit #AFFB-12, specimen D, Table I) was run by emission with the Fourier instrument. For this purpose the tubing on which the deposit had been formed was flattened and the backside attached to a small cartridge heater. The front surface was then placed in the focal plane of the inlet system. Figure 4 shows the spectrum in the 630 - 1230 cm^{-1} range, Figure 5 in the 1275 - 1850 cm^{-1} range and Figure 6 in the 2600 - 3600 cm^{-1} range. It is in the nature of our Fourier instrumentation that separate runs must be made to cover separate spectral ranges. The instrument was flushed with nitrogen in the intermediate spectral range, but atmospheric absorption bands still show in the 1400-1700 cm^{-1} region. The best defined emission features of this specimen in the low frequency region (Figure 4) are the 700 cm^{-1} peak, shoulders near 740 and 760 cm^{-1}, a shoulder at 870 cm^{-1}, peaks at 930 and 1000 cm^{-1} and shoulders at 1050 and 1110 cm^{-1}. All of these bands are consistent with subsitituted monocyclic aromatics through dicyclics cannot be excluded. In the intermediate frequency region (Figure 5) the initial downward slope is caused by diamond window absorption, which is particularly strong from

1650 cm^{-1} upwards (to about 2200 cm^{-1}). The broad band near 1600 cm^{-1} is also consistent with the presence of aromatics. Two bands at 1755 and at 1720 cm^{-1} could be caused by carbonyl, such as acids and esters. The high frequency region (Figure 6) contains some very pronounced emission bands, notably at 2800, 2910, 3120, 3230, and 3480 cm^{-1}. The first two frequencies are probably caused by CH_2 and CH_3 stretches. 3120 cm^{-1} is again characteristic of aromatics and the high frequency bands could be assigned to amides and phenols. The 3480 cm^{-1} band is, of course, the same as the one observed by reflection. External reflection and absorption should correspond in frequency whenever both are observed, but the relative band intensities can be quite different.

In summary, therefore, it would appear that this sample, specimen D (Table I) is likely aromatic, containing some phenols and/or nitrogeneous materials as well.

The emission spectra of specimen A (KLM PFA 60701), were obtained from the scraped powdered deposit run as a sandwich between a heated flat metal surface and a potassium chloride window. The three regions, 630 cm^{-1} - 1230 cm^{-1}, 1275 - 1850 cm^{-1}, and 2600 - 3600 cm^{-1}, are shown in Figures 7, 8, and 9. The low frequency region of Figure 7 is similar to that of Figure 4, but less pronounced. The intermediate frequency region is also similar, but with some shifts of peak frequencies. On the other hand, the high frequency region (Figure 9) appears to be quite different from that of specimen D (Figure 6). Closer comparison of Figures 6 and 9 shows that some of the emission bands of Figure 6 are totally or partly inverted in Figure 9, notably the bands peaking at about 2800 cm^{-1} and at 3230 cm^{-1}. This phenomenon is not an unusual occurrence with powdered samples, where scattering, reflection, and re-absorption can be superimposed on emission. This example shows that the running and interpretation of emission spectra is not as straight-forward as that of absorption spectra. A spectrum, such as that of Figure 9 could be interpreted as just noise, and indeed, a thicker layer of the powder would probably have only a noise spectrum.

Specimen A is thus similar to specimen D, also primarily aromatic, but probably of lower concentrations.

Figures 10, 11, and 12 are spectra obtained from the TF-30 Engine S/N 585 No. 8 fuel manifold (specimen C, Table I). The deposits were shaken out by light tapping to the outside and then ground in a mortar. The data were taken by emission from the deposit placed on a heat cartridge. Similar spectra could be obtained directly by focusing with the Cassegrainian microscope objective, but their quality was not as good. There is little doubt, however, that more effort would produce equivalent spectra without scraping and grinding, which are, as stated not very satisfactory analytical procedures.

Comparison of Figures 4 and 10 (low frequency range) shows the presence of most of the same peaks in the both figures, but of more minor peaks in the latter. The intermediate frequency range, Figure 11, is rather different from that of Figure 5, note that the 1730/50 doublet has

been replaced by a broader band centered about a slight dip near 1740 cm^{-1}. The pronounced band near 1590 cm^{-1} of Figure 5 has almost vanished. These results are consistent with a higher oxygen content (less hydrocarbon, possibly more carbonyl).

Figure 12 is the high frequency spectrum of Specimen C. It is quite different from that of Figure 6. The 2810 cm^{-1} is now absent almost entirely and only absorptions are shown near 3190 and 3250 cm^{-1}. Amides are a good possibility. Such an assignment would be consistent with the higher oxygen and nitrogen content of this material compared to those of the others (Table I).

V. DISCUSSION

- The elemental analyses show the highest oxygen and nitrogen
contents for specimen C, the highest carbon and hydrogen content for
specimen D. The infrared emission spectra are consistent with these
analyses, showing the strongest aromatic bands for specimen D and
possible amide groups for specimen C.

While the assignments of the infrared bands were stated to be
consistent with di- and trisubstituted monocyclic rings, they are
also consistent with analogous condensed rings. For example, a pyrene
structure of the type
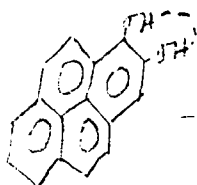
is entirely consistent with the infrared emission spectra as well as
as with the elemented analysis. It is well-known that tars and as-
phaltenes of similar composition occur in petroleum. The ultraviolet
absorption or fluorescence spectrum could help clarify the structural
assignments.

The differences between specimens A, C, and D,are undoubtedly
related to the differences between JP-4 and JP-7.

Years ago the author participated in a study of the composition
and role of petroleum asphaltenes and various tars. Their strong
adhesion to surfaces is, of course, well known. The purpose of this
study was the development of direct and fast methods of analysis of
jet fuel deposits. Our infrared emission spectrophotometry is evi-
dently such a method, so that the purpose has been fulfilled. Beyond
that, it seems that the deposits are related to my old friends, the
asphaltenes. Obviously, it is dangerous to speculate on the basis of
so few samples. Further work is clearly indicated.

VI. ACKNOWLEDGEMENT

TABLE I*

SOME PROPERTIES OF JET FUEL DEPOSITS

| Sample	Description	Elemental Analysis	
		Element	%
A	"Carbon" deposit derived from JP-4 fuel scraped from the valve cavity of a fuel nozzle from a CF6-50A (G.E.) KLM engine. The engine had been operated 3000 hours with a throughput of 660,000 pounds of fuel. The total weight of deposit was 0.4992 g, corresponding to 1.7 parts of "carbon" per billion parts of fuel. (KLM PFA 60701, powder)	C H N O Residue Sum	50.3, 55.9 2.39 0.56, 0.78 20.5 20.9 95.1
B	Fuel "gum" from JP-4 fuel of same engine	C H N O Residue Sum	
C	TF-30 Engine S/N 585 No. 8, Fuel Manifold, deposit from inner wall shaken out by light tapping to outside and then pulverized in a mortar	C H N O Residue Sum	56.4, 55.9 3.05, 2.88 1.38, 1.36 26.5, 26.5 5.60, 6.70 92.9, 93.3
D	Jet fuel deposit #AFFB-12-68 from JP-7 fuel, deposit on inner surface of tubing. This deposit had the following atom ratios: H/C 0.63, 0.61, N/C 0.010, 0.011, O/C 0.10, 0.10, S/C 0.003, 0.003	C H N O S Residue Sum	79.7, 80.4 4.2, 4.1 0.9, 1.0 10.9, 10.6 0.69, 0.79 0.1, 0.4 96.3, 97.0

* data supplied by Dr. Hazlett of NRL

FIGURE CAPTIONS

1. Optical diagram of internal reflection attachment to infrared spectrometer (VRA)

2. VRA with specular reflection attachment

3a. Reflectance spectrum of specimen D, high wavenumbers
 b. Reflectance spectrum of specimen D, low wavenumbers

4. Emission spectrum of specimen D, at $50^{\circ}C$, low frequency range

5. Emission spectrum of specimen D, at $50^{\circ}C$, intermediate frequency range

6. Emission spectrum of specimen D, at $65^{\circ}C$, high frequency range

7. Emission spectrum of specimen A, at $65^{\circ}C$, low frequency range

8. Emission spectrum of specimen A, at $65^{\circ}C$, intermidiate frequency range

9. Emission spectrum of specimen A, at $65^{\circ}C$, high frequency range

10. Emission spectrum of specimen C, at $65^{\circ}C$, low frequency range

11. Emission spectrum of specimen C, at $65^{\circ}C$, intermediate frequency range

12. Emission spectrum of specimen C, at $65^{\circ}C$, high frequency range
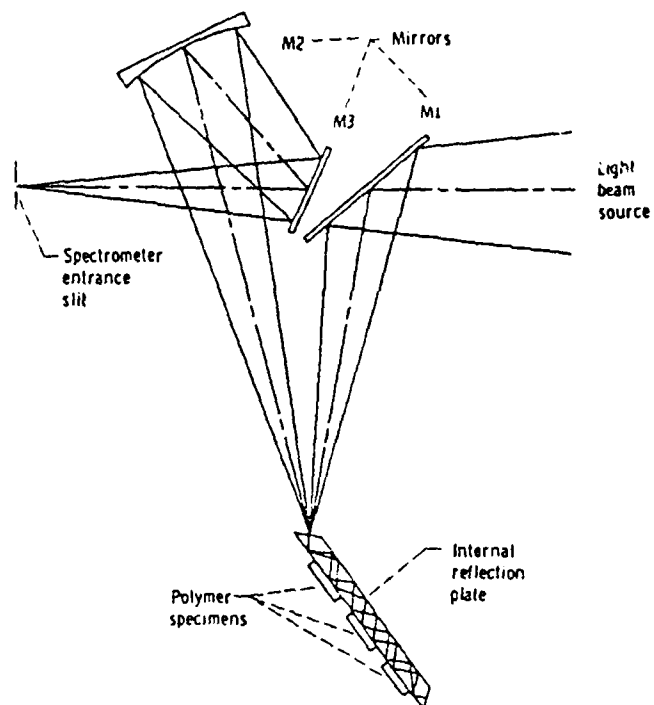
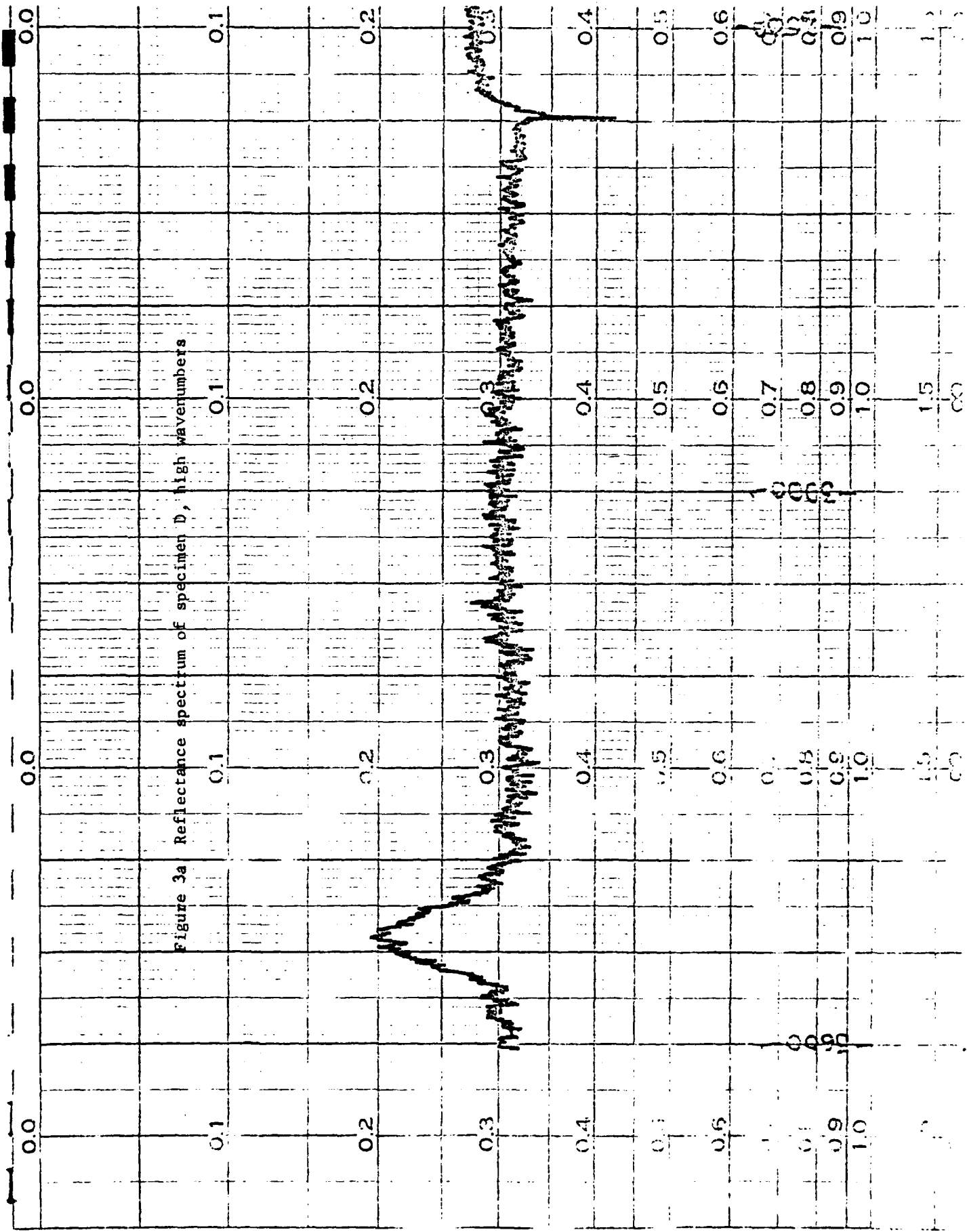Figure 1. - Optical diagram of internal reflection attachment to infrared spectrometer.
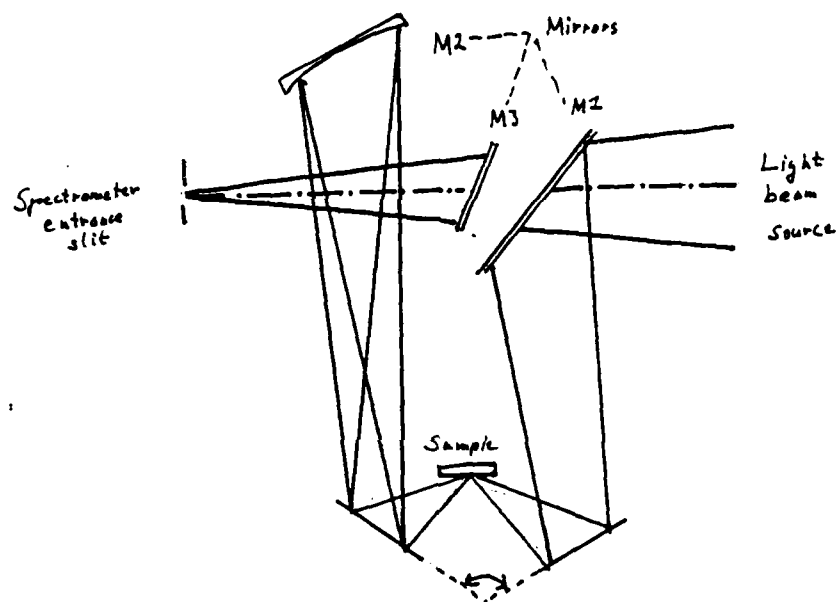
Figure 2   VRA with specular reflection attachment

Figure 3a Reflectance spectrum of specimen D, high wavenumbers
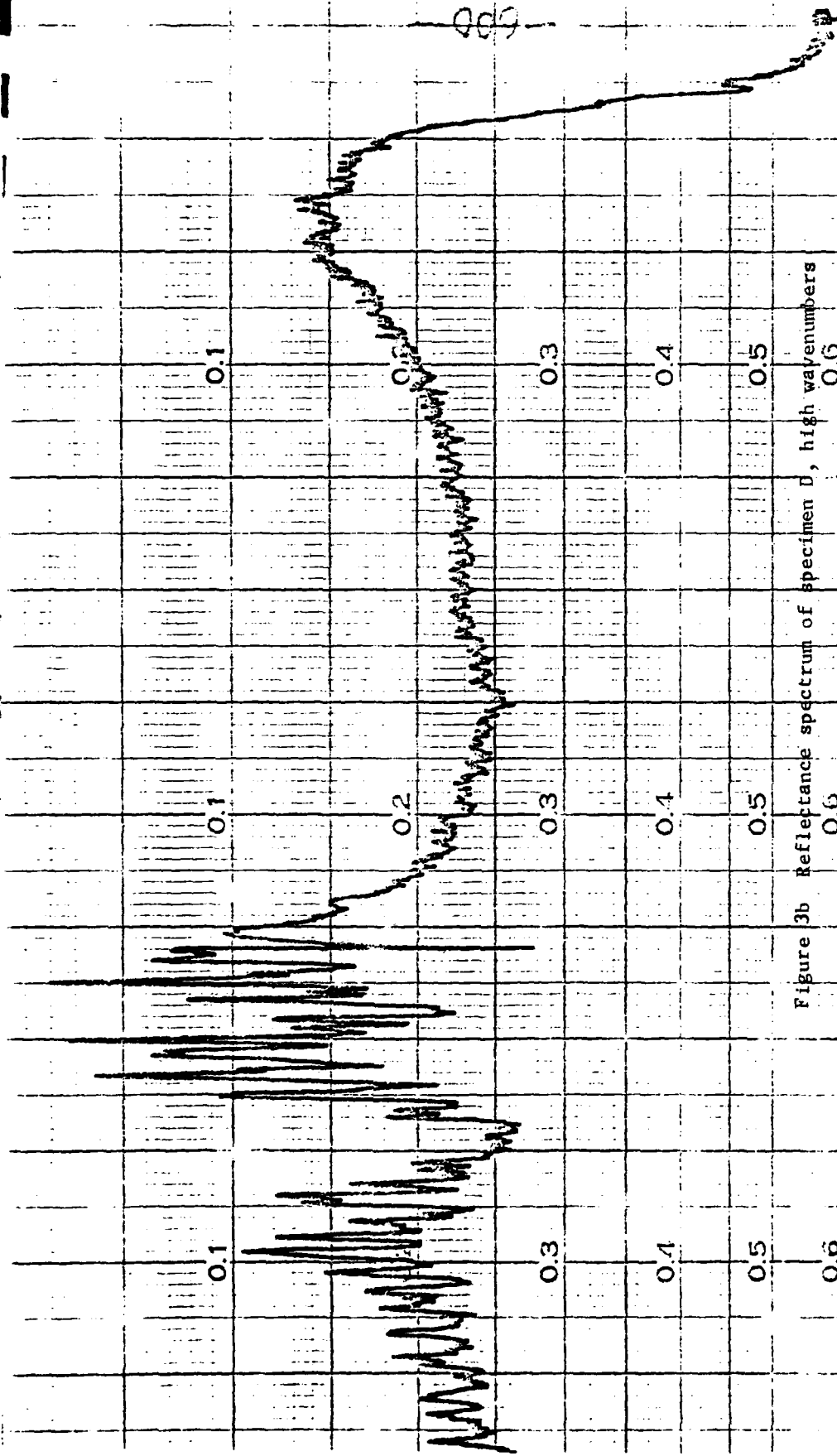
Figure 3b Reflectance spectrum of specimen D, high wavenumbers
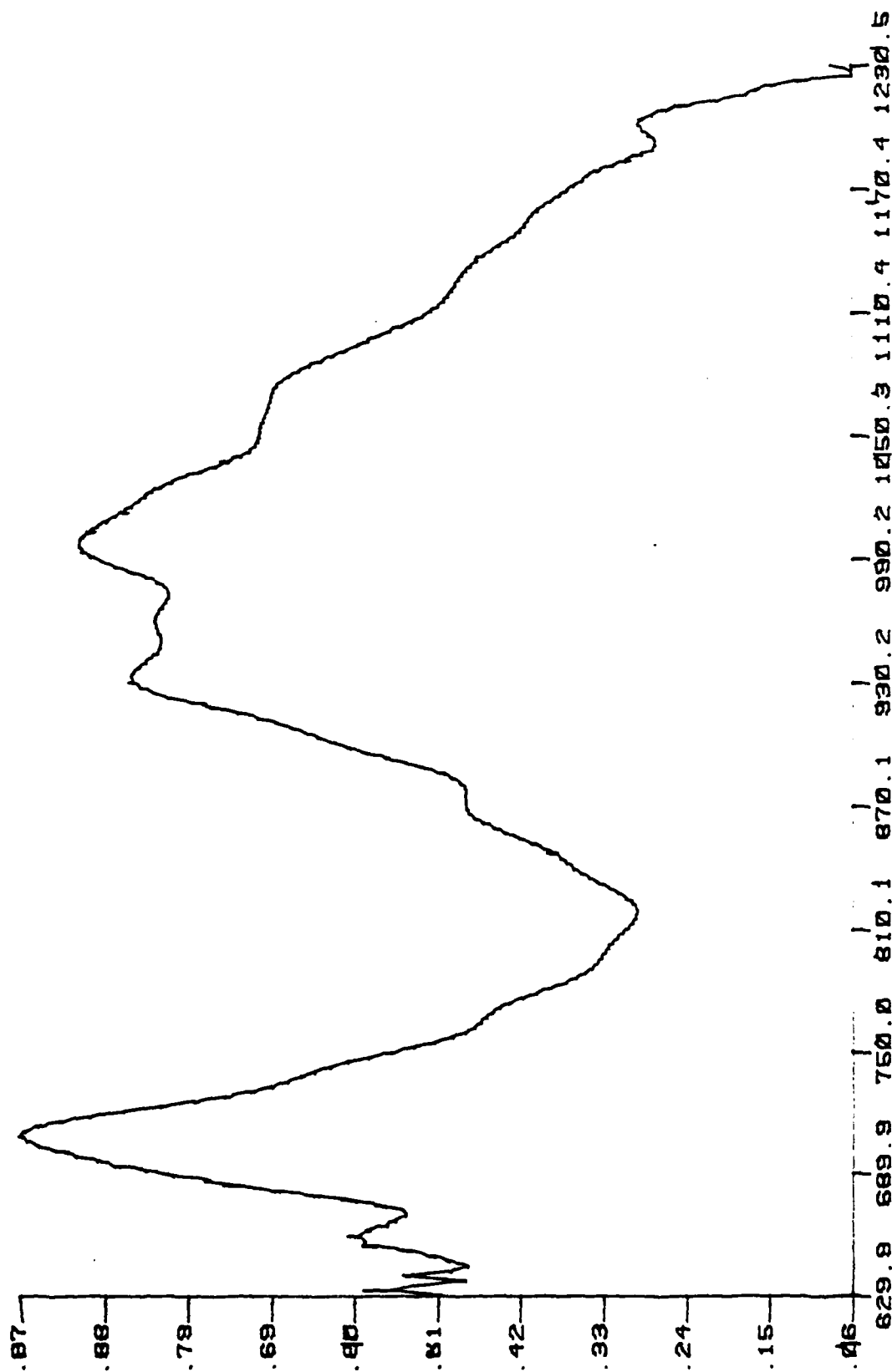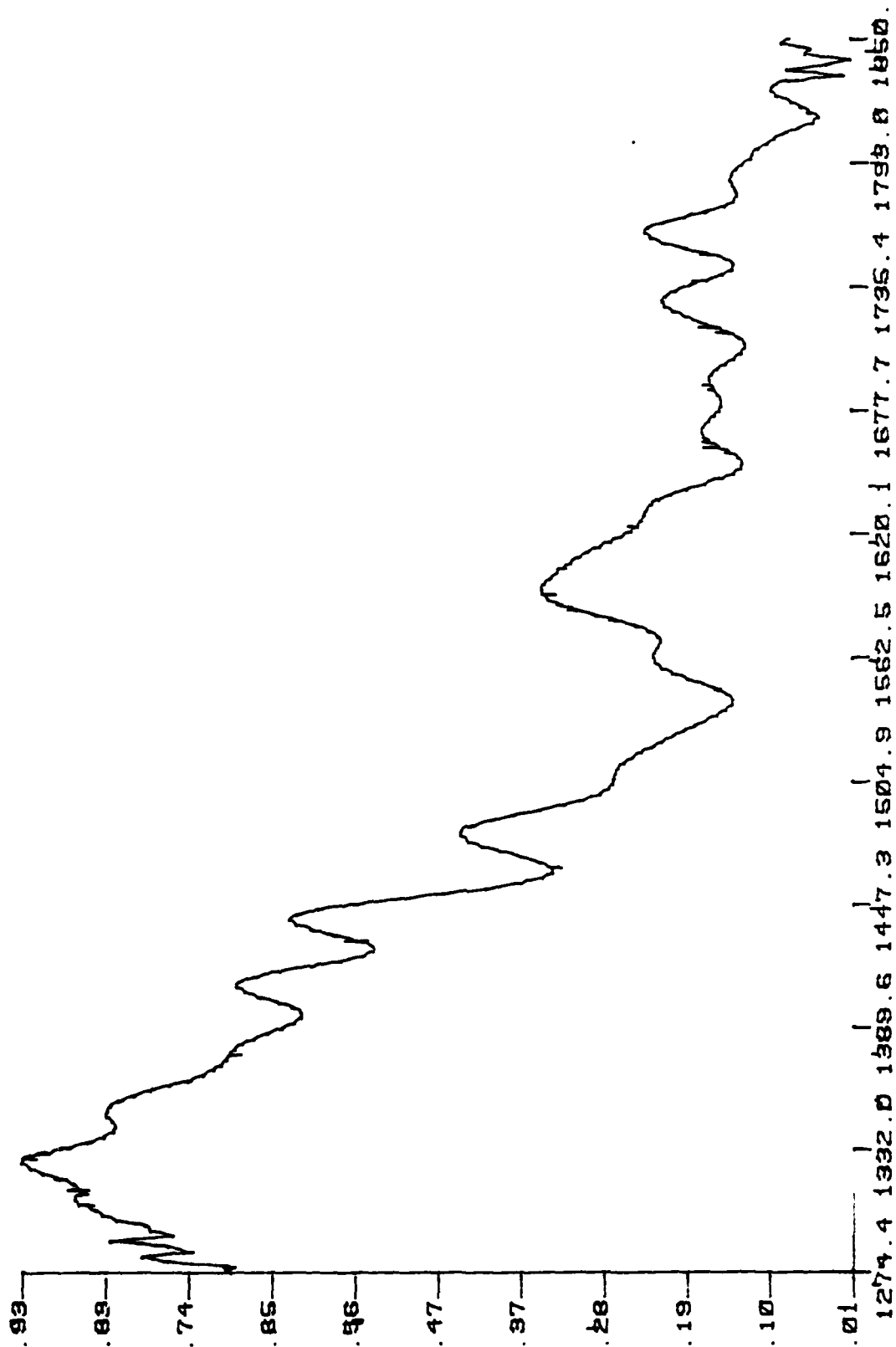
.87
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629.8 689.9 750.0 810.1 870.1 930.2 990.2 1050.3 1110.4 1170.4 1230.5

SPECTRUM NUMBER: 230

Figure 4 Emission spectrum of specimen D, at 50°C, low frequency range
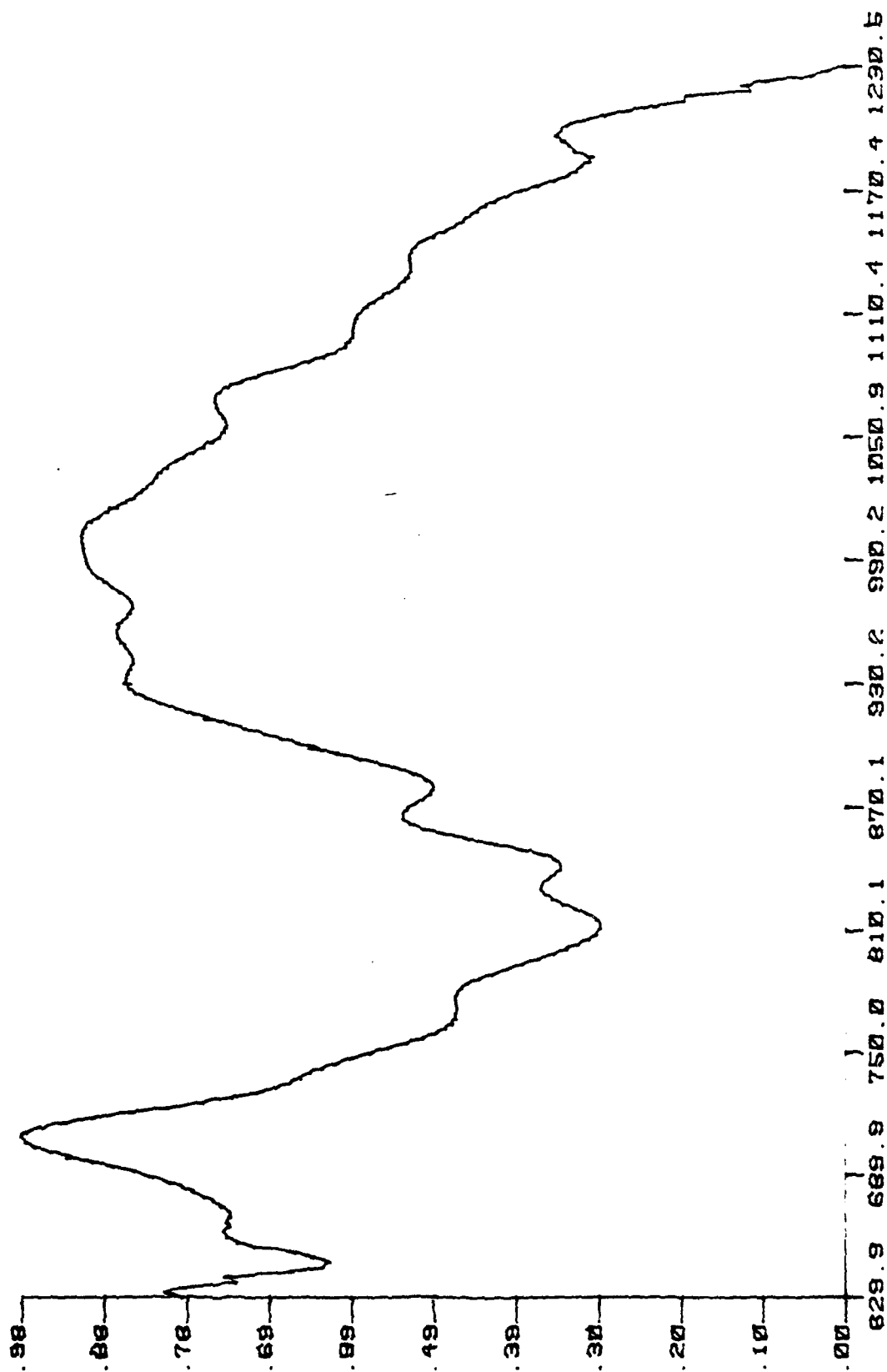
SPECTRUM NUMBER: 231

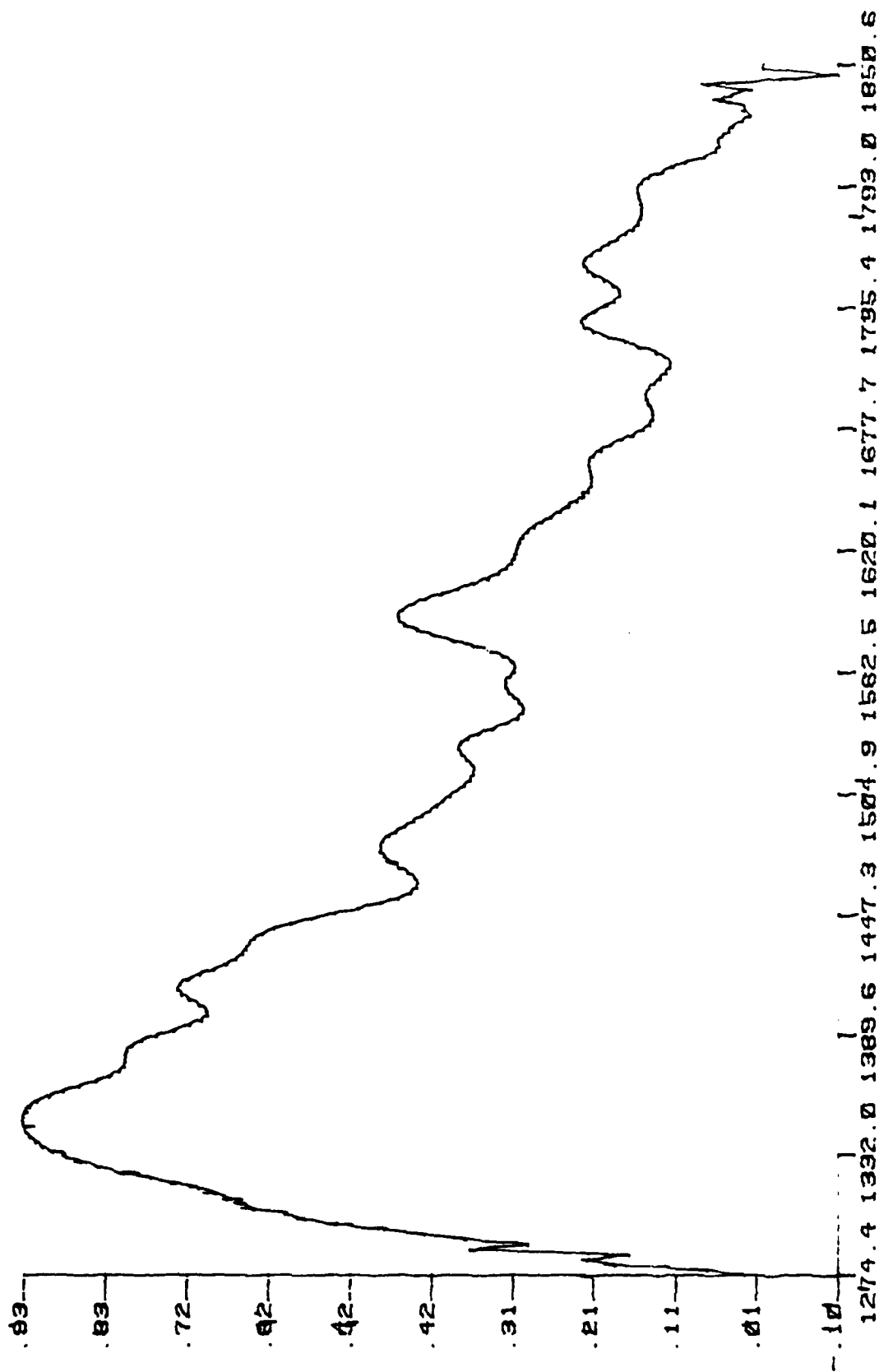Figure 5   Emission spectrum of specimen D, at 50°C, intermediate frequency range
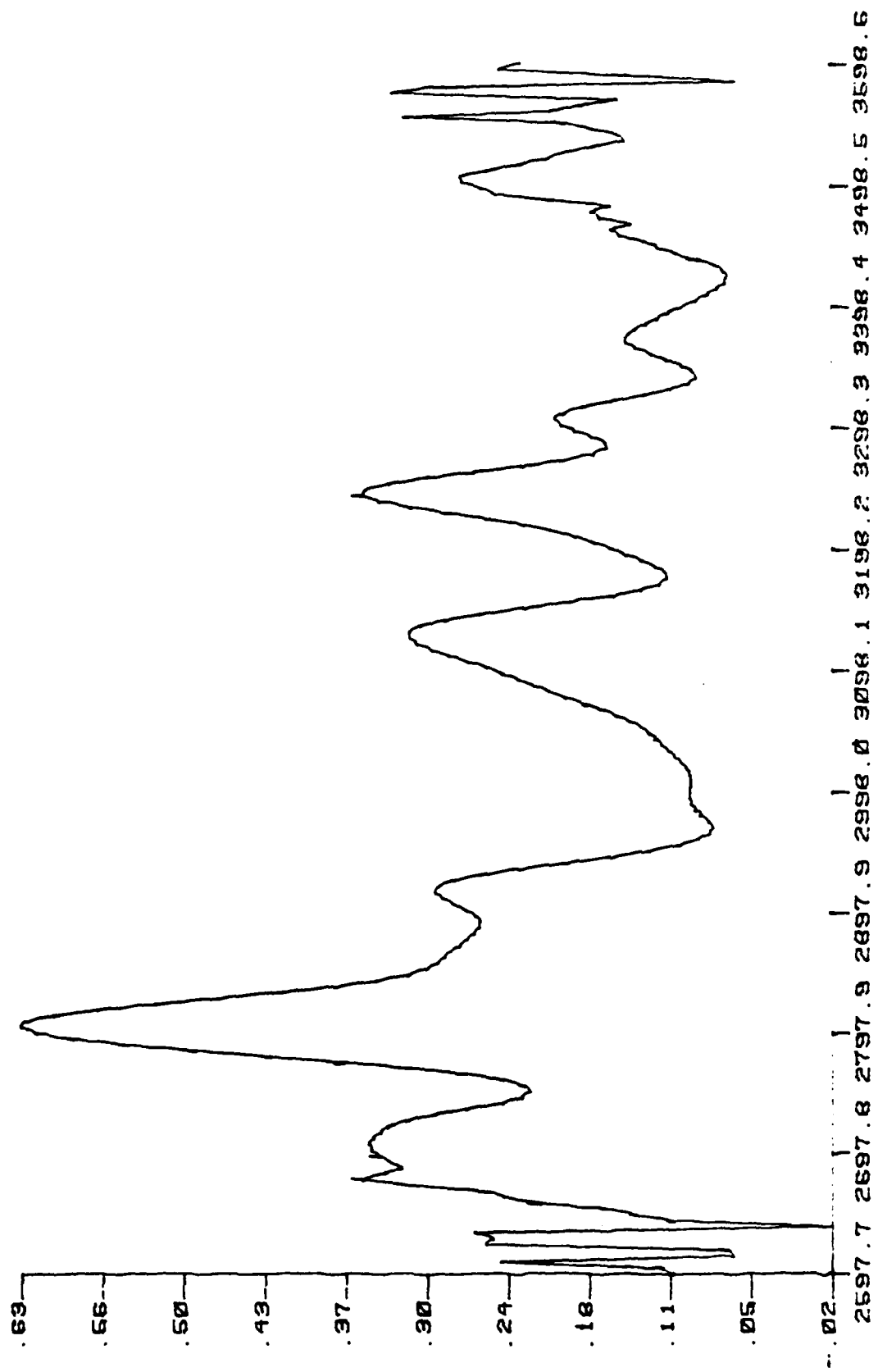
SPECTRUM NUMBER: 232

Figure 6 Emission spectrum of specimen D, at 65°C, high frequency range

SPECTRUM NUMBER: 234

Figure 7 Emission spectrum of specimen A, at 65°C, low frequency range

SPECTRUM NUMBER: 235

Figure 8 Emission spectrum of specimen A, at 65°C, intermediate frequency range

SPECTRUM NUMBER: 233

Figure 9 Emission spectrum of specimen A, at 65°C, high frequency range

SPECTRUM NUMBER: 234

Figure 10 Emission spectrum of specimen ., at 65°C, low frequency range

SPECTRUM NUMBER: 237

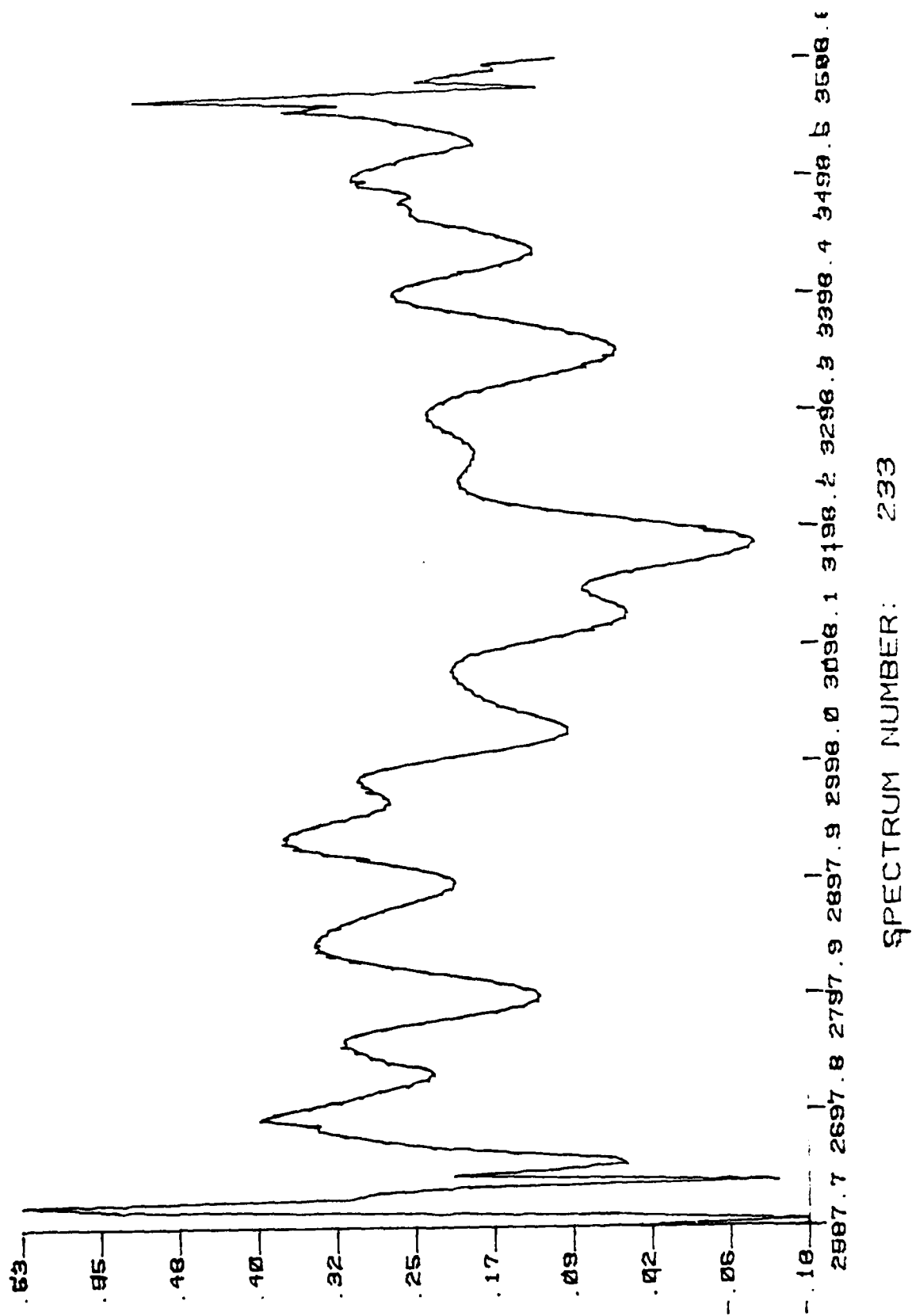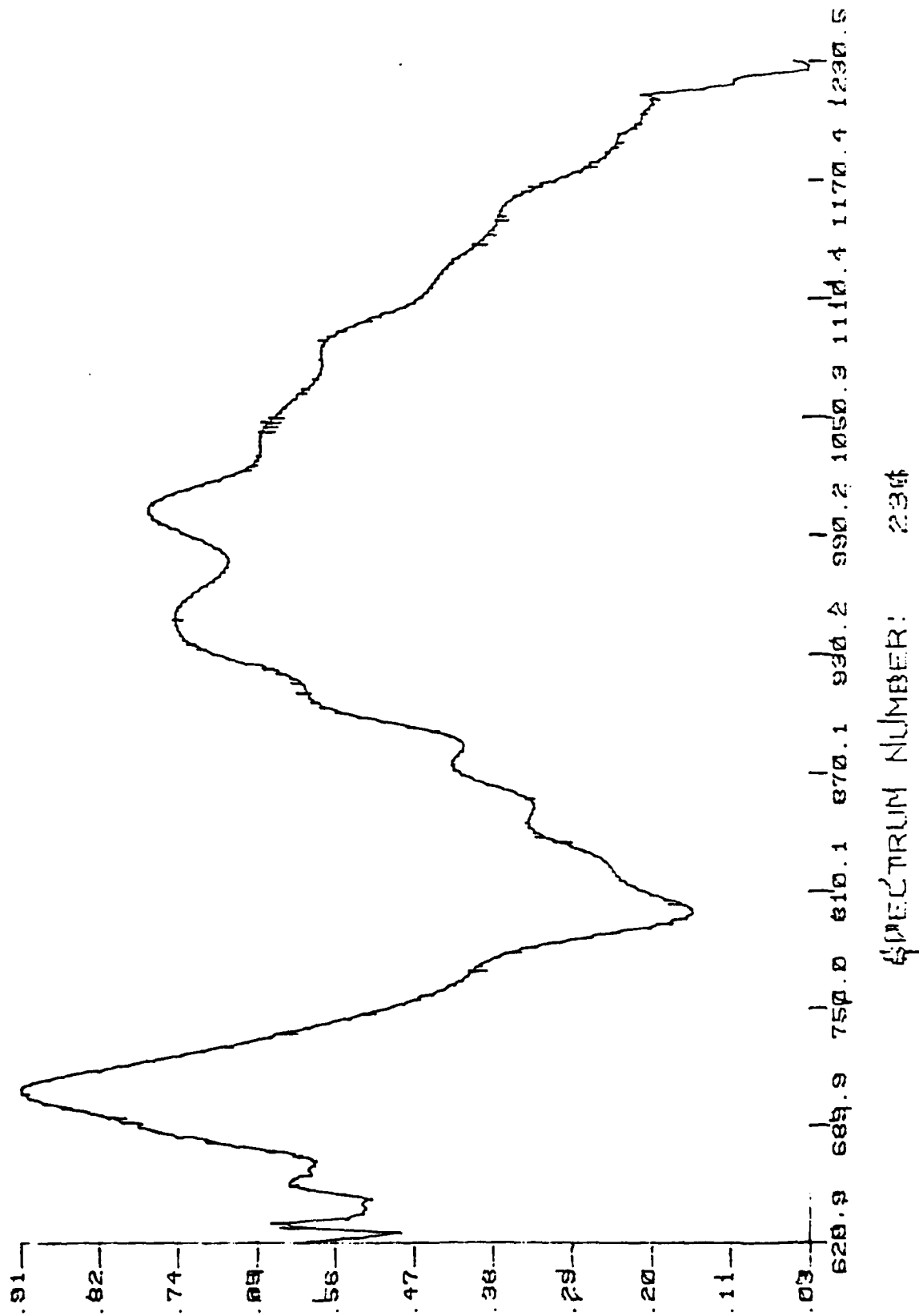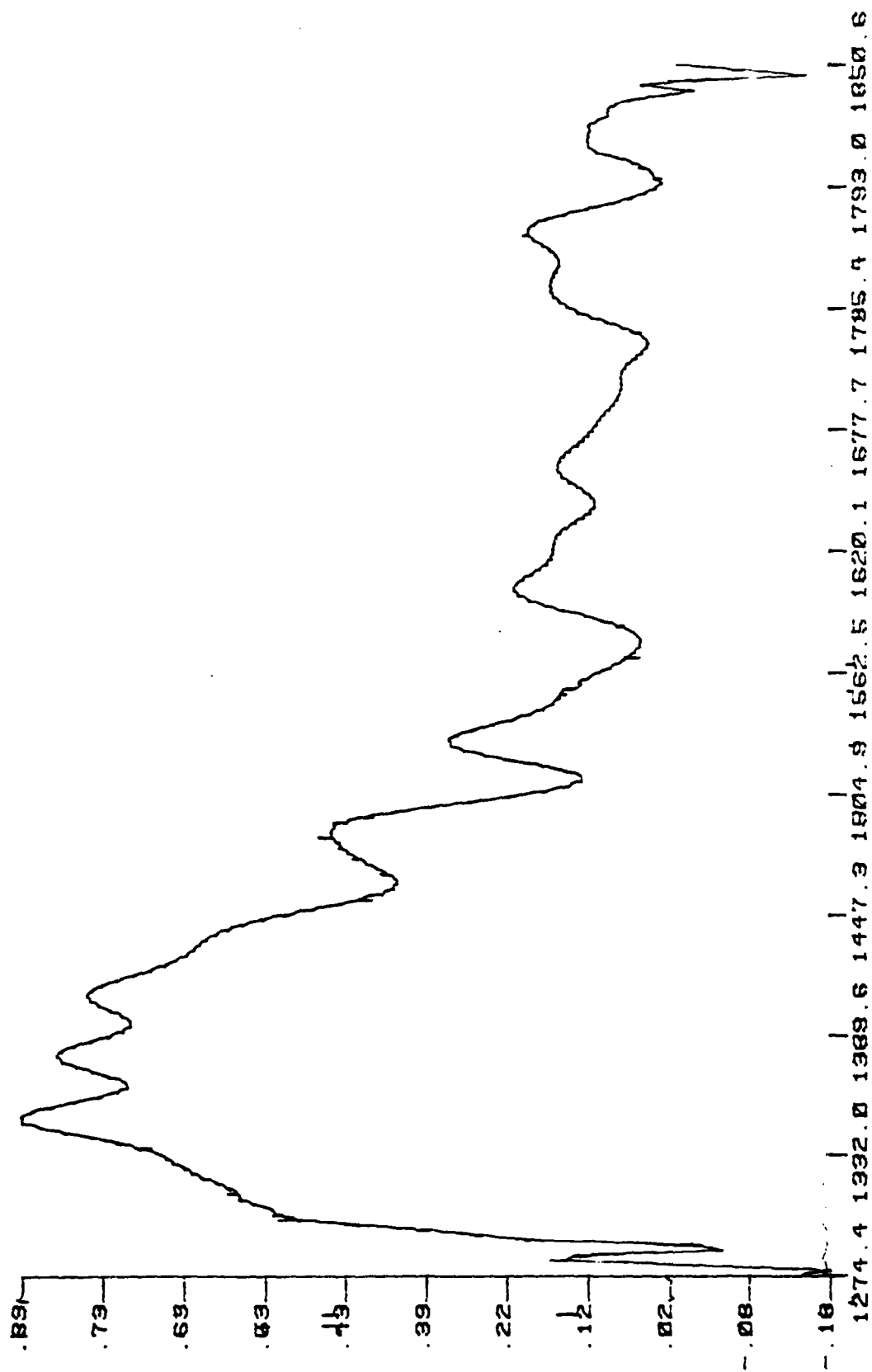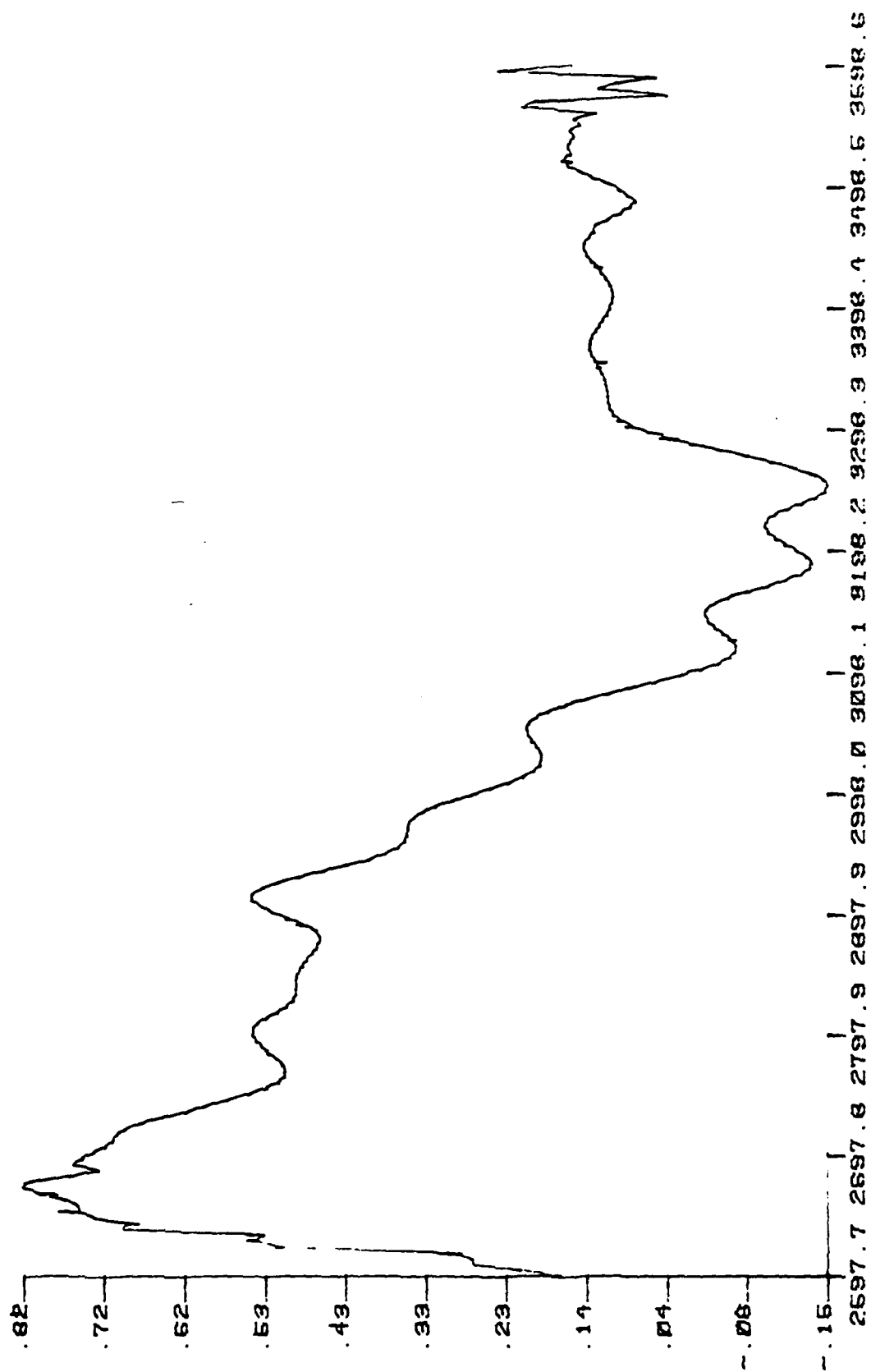Figure 11 Emission spectrum of specimen C, at 65°C, intermediate frequency range

SPECTRUM NUMBER: 238

Figure 12 Emission spectrum of Specimen C, high frequency range

APPENDIX   I

Recent paper describing our infrared Fourier emission microspectrometer.

(pages 395, 401, and 402 only)

FOURIER EMISSION INFRARED MICROSPECTROPHOTOMETER FOR SURFACE ANALYSIS—I. APPLICATION TO LUBRICATION PROBLEMS

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Rensselaer Polytechnic Institute, Troy, New York, U.S.A.

Abstract—A Beckman-RIIC FS-720 far-infrared interferometer was converted into an emission microspectrophotometer for surface analysis. To cover the mid-infrared as well as the far-infrared the Mylar beamsplitter was made replaceable by a germanium-coated salt plate, and the Moiré fringe counting system used to locate the moveable Michelson mirror was improved to read 0.5 μm of mirror displacement. Digital electronics and a dedicated minicomputer were installed for data collection and processing. The most critical element for the recording of weak emission spectra from small areas was, however, a reflecting microscope objective and phase-locked signal detection with simultaneous referencing to a blackbody source.

An application of the technique to lubrication problems is shown.

INTRODUCTION

The recording of infrared emission spectra from moderately heated surfaces was one of the first achievements of infrared interferometry when it was introduced in the 1960's. Low[1] showed spectra of organic acids on flat metal surfaces and was able to interpret them in terms of salt formations. Griffith[2] showed spectra of greases on metallic mirrors and noticed that the spectral contrast was very much better for exceedingly thin (presumably less than 1 μm thick) coatings than for thicker ones, presumably because of self-absorption. Much further work has since been reported; a recent article by Bates[3] provides a good summary.

In all of these studies the radiating area was essentially unlimited. True, sources larger than a cm² cannot be handled by the ordinary Fourier spectrometer, but this area is usually large enough to provide adequate signal even at temperatures as low as 35°C. In many fields of study spatial resolution is necessary: for example, when a lubricated ball bearing is examined during operation, the diameter of the contact area is always less than 500 μm and usually less than 100 μm. Biological structures in tissues are even smaller. Corroded areas on metal surfaces and inclusions in minerals and fibers are other examples requiring the analysis of small areas.

The apparatus described in the following sections was assembled to meet this need. Available components were used where possible to minimise cost. The use of a slow-scanning interferometer may appear to be old-fashioned nowadays, when ultrafast, real-time instruments are marketed. However, when the large number of scans required for adequate signal-to-noise ratio is taken into account, the slow-scanning instrument is really not slower and, furthermore, as Griffith[4] pointed out, for very weak signals slow-scanning can have advantages over fast-scanning. In any case, workers having a fast-scanning Fourier interferometer at their disposal will have little difficulty in modifying their instrumentation for measurements similar to ours. Some aspects of an application to a lubrication problem will be shown.

THEORY

As was pointed out by Hordvik[5] infrared emission is probably the most sensitive method for the detection of weak infrared bands, especially in thin films. Band absorption coefficients as low as $10^{-5}\,cm^{-1}$ can be measured routinely and as low as

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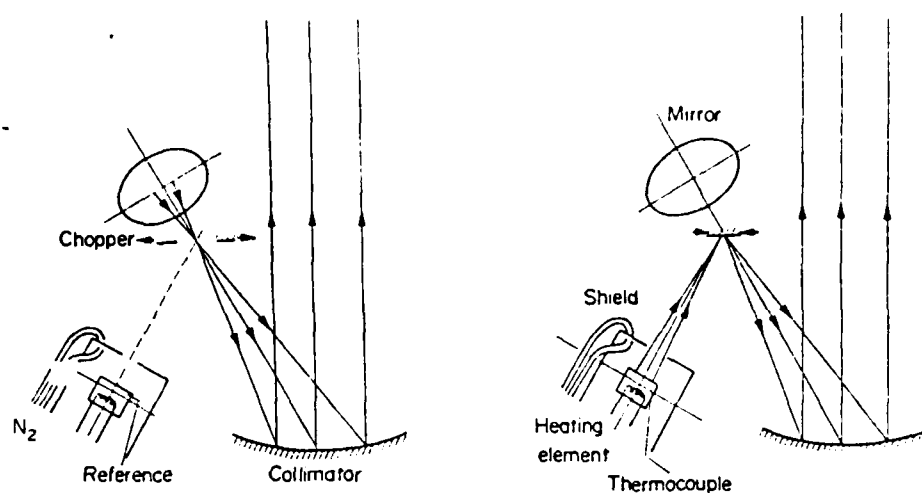*To whom correspondence should be directed

Fig. 3. Chopper and reference assembly.

the case of the bearing the graybody radiation from the solid surfaces can exceed the discrete radiation from the lubricating film by orders of magnitude in overall intensity. If the total radiation were detected and analysed simultaneously, the discrete radiation would be lost in the noise level. (The discrete radiation of the lubricant, which gives rise to an emission spectrum that is equivalent to an absorption spectrum is, of course, our main interest. Discrete emission bands appear at the absorption frequencies for, in a simple-minded model, the ball's emissivity is higher there than at other frequencies because the covering fluid constitutes a dark surface layer at absorptions.) Hence it is necessary to oppose the continuous radiation by a blackbody source. This is done by the chopper and reference shown in Fig. 3. The chopper, consisting of a pair of reflecting tuning fork tines, vibrating about the collimator focus in a plane oriented at 45° with respect to the direction of the source radiation, alternately introduces source and reference radiation into the spectrometer. The reference is a block of brass, heated by a cartridge heater, which is shielded by air-cooled aluminium sheet except for a conical opening directed toward the chopper. The temperature of the reference is so adjusted that the average amplified signal level from the lock-in amplifier is essentially zero.

If it were possible for us to obtain complete balance between the blackbody source radiation and the reference radiation over a wide wavelength region—e.g. by inserting optical filters of different passbands and continuing to maintain balance—the reference temperature and total radiance would be equal to that of the blackbody part of the source. In fact, it is only possible to adjust the radiance over a narrow spectral region, the blackbody slopes being different because of their different temperatures. Because of that the level of the reference radiation is sometimes between the peak and the bottom of an emission band (Fig. 4). Since only absolute values of the amplitude of the difference between source and reference are calculated by the Fourier transformation, distorted spectra can result as also shown in Fig. 4. In fact, if the level of the reference radiation is above the peak of an emission band, the emission band will come out inverted and appear as an absorption band. What makes this artefact so difficult to find at times is that correct emission bands can appear in one frequency range of the spectrum, but inverted (looking like absorption) bands in another. We have learnt to live with this phenomenon by (i) optically limiting the frequency range as much as possible and thereby reducing the slopes and (ii) by obtaining duplicate spectra with slightly different reference temperatures. Thus, Fig. 5 shows an apparent absorption band at $690 \, \text{cm}^{-1}$. Figure 6, obtained with a slightly lower reference temperature, shows the correct emission band.

gathering power is superb and the Golay detector is linear over the entire frequency range available, viz. 50–4000 cm^{-1}. The difference between our operation and that usually encountered in absorption spectrophotometry should be emphasised: we are looking for signals from a weak source which, moreover, we must separate into a continuous background and discrete emission bands, while the absorption spectroscopists are looking for small differences of intensity in very large signals from a very hot source.

(ii) Emission optics

The small radiating area and its low temperature require first of all a collecting lens so that radiation is introduced into the apparatus from a reasonably large solid angle. For this purpose the Cassegrainian reflecting objectives (lenses) of Beck Ltd are ideal; they come with different numerical apertures and magnifications, covering fields of view varying from 10 μm to 1250 μm in diameter for essentially equal radiation throughput. By putting different lenses below the diamond window in the bottom of a cup filled with lubricant it was possible to gather radiation from different portions of the conjunction region with a loaded ball rotating (sliding) above this window in the cup. The radiation was generated by the viscous friction of the lubricant separating diamond and ball surfaces. Here the lubricant layer had to be centered about the optic axis of the lens and exactly in its focal plane (Fig. 2). Other specimens could be examined by placing them directly on the diamond window.

The collecting lens directs the source radiation downward to a 45° plane mirror, which brings it into the horizontal plane of incidence of the Michelson interferometer. An enlarged image of the source is formed a short distance downstream of this mirror. This image is in the focal plane of the interferometer's collimator and constitutes the effective source for the detector. Another image is formed just ahead of the detector, where an iris diaphragm is used to select portions of the object for analysis.

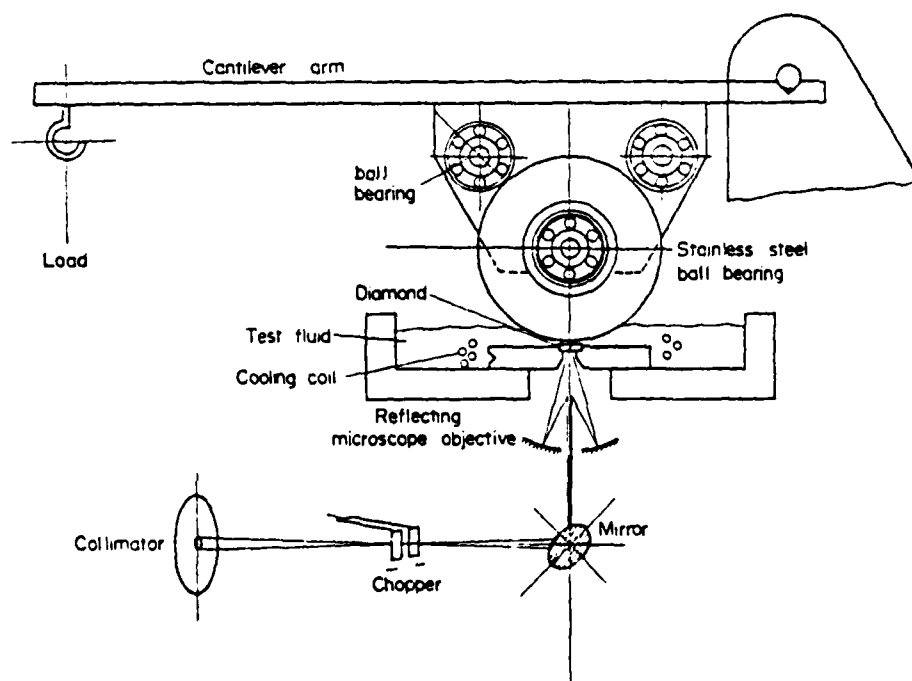The radiation from the lubricating layer in a bearing or from other weak sources is invariably superimposed on a background of greybody or blackbody radiation. In

Fig. 2. Mockup bearing assembly.